Nano Vision, Vol.1 (2), 68-73 (2011)

Synthesis of Cu_2O Nanoparticles Using Simplified Polyol Process

K. KARTHIK[a] and S. PONNUSWAMY[b]

[a]Department of Chemistry, Kumaraguru College of Technology (Autonomous), Coimbatore – 641 049, Tamilnadu, India.
[b]Department of Chemistry, Government Arts College (Autonomous), Coimbatore-641 018, Tamilnadu, India.

ABSTRACT

Cuprous oxide (Cu_2O) nanoparticles have been synthesized by a simplified polyol mediated thermolysis process without adding any surfactant or capping agent. The average size of the particles is in the range of 17-57 nm calculated from XRD spectrum using Scherrer formula. The effects of reaction temperature, concentration of the chemicals and reaction time over the size of the Cu_2O nanoparticles have been studied. The SEM images revealed that the Cu_2O particles are in the cauliflower like structure.

Keywords: Cu_2O nanoparticles, polyol process, cauliflower-like structure.

INTRODUCTION

Cuprous oxide (Cu_2O) shows a unique electronic structure for applications[1-15], in particular those related to chemical and photochemical processes such as the splitting of water under visible light irradiation[1-6]. The electron-correlation effects on the electronic structure of transition metal compounds and the high Tc super conductors can be studied using both Cu_2O and CuO[7]. It has been found that Cu_2O microspheres can be used as the negative electrode material for lithium ion batteries[8]. In addition, Cu_2O has attracted the researchers due to its importance in P-type semiconductors[9,10]. The fabrication of metal oxide semiconducting nanomaterials with large surface area to volume ratios for gas sensor applications is currently a major focus on nanoscience and nanotechnology[11-15].

Many differently shaped Cu_2O nanostructures have been synthesized using various approaches such as electrodeposition[16,17], thermal relaxation[18], sonochemical method[19], vaccum evaporation[20], solvothermal method[21] and liquid phase reduction of metal salts[22,23]. Disk-like Cu_2O nanostructures have also been synthesized using polyol process by adding PVP K-30 as a capping agent[7]. Herein, we report a simplified polyol

process to synthesize the Cu_2O nanoparticles without adding any surfactant or capping agent. In this process, the polyol (polyethylene glycol-400) could serve as both solvent and reducing agent.

EXPERIMENTAL

A typical procedure is as follows: 50 ml of anhydrous polyethylene glycol-400 (PEG-400) was mixed with 3 g of copperacetate monohydrate salt in a round bottom flask equipped with a water condenser. The reaction mixture was green in colour and it was allowed to reflux at ~ 360°C for 3 hours. The green coloured reaction mixture was changed into reddish-brown colour. Finally, the resultant sample was filtered, washed with acetone, dried in air before further characterization. The reddish colour of the sample reveals the presence of Cu_2O particles.

RESULTS AND DISCUSSION

The crystal structure and average particle size of the Cu_2O particles were analysed by X-ray diffraction (XRD). The morphology of the Cu_2O particles was analysed by scanning electron microscopy (SEM). The XRD pattern of the prepared Cu_2O particles is shown in Figure 1. It is observed that there are much broader and less intense peaks in the XRD spectrum, owing to particle size broadening, which occurs when a sample is made up of very small crystallites. The broadness of the peak can be used to calculate the average crystalline size of the Cu_2O particles using the Scherrer's formula[24], ($D = 0.94\lambda/ \beta\cos\theta$), where 0.94 is a constant value known as shape factor, λ is the wavelength of the X-rays, β is the full width at half maximum of the diffraction peaks and θ is the angle of diffraction. The XRD pattern of the as prepared Cu_2O particles is in good agreement with the reported XRD pattern of Cu_2O particles[7].

The average crystalline size of the Cu_2O particles calculated using the Scherrer's formula for the Cu_2O sample shown in Figure 1 is in the range of 29-37 nm. The peaks at 2θ values of 36.365° and 42.246° are in good agreement with the cubic Cu_2O phase and correspond to 110 and 200 lattice planes of standard crystalline Cu_2O, respectively[7]. The morphology was analysed using SEM and it shows that the Cu_2O particles are cauliflower like shapes (Figure 2).

In order to study the effect of reaction temperature, concentration and reaction time over the size of the Cu_2O nanoparticles, the reactions were performed at different reaction conditions and fifteen Cu_2O samples have been synthesized and the average size of the particles is in the range of 17 – 57 nm. The results along with the reaction conditions are presented in Table 1, 2 & 3.

The results of the XRD spectra revealed that the increase in reaction temperature decreases the size of the particles up to 250°C. When we increase the concentration ratio of PEG-400 to copperacetate monohydrate, the size of the particles also increases. Hence the concentration of the constituent chemicals can control the size of Cu2O particles. The size of the Cu_2O particles is insensitive to the reaction time above 2 hrs to 6hrs.

In summary, cauliflower shaped Cu_2O nanoparticles have been synthesized by a simplified polyol process without adding any surfactant or capping agent. The average size of the particles lies in the range of 17-57 nm calculated using Scherrer's formula. XRD, SEM investigations on these Cu_2O particles have been carried out. Though the reaction time may not have a greater influence on the size of the Cu_2O particles, the reaction concentration does have an influence on the size of the Cu_2O particles.

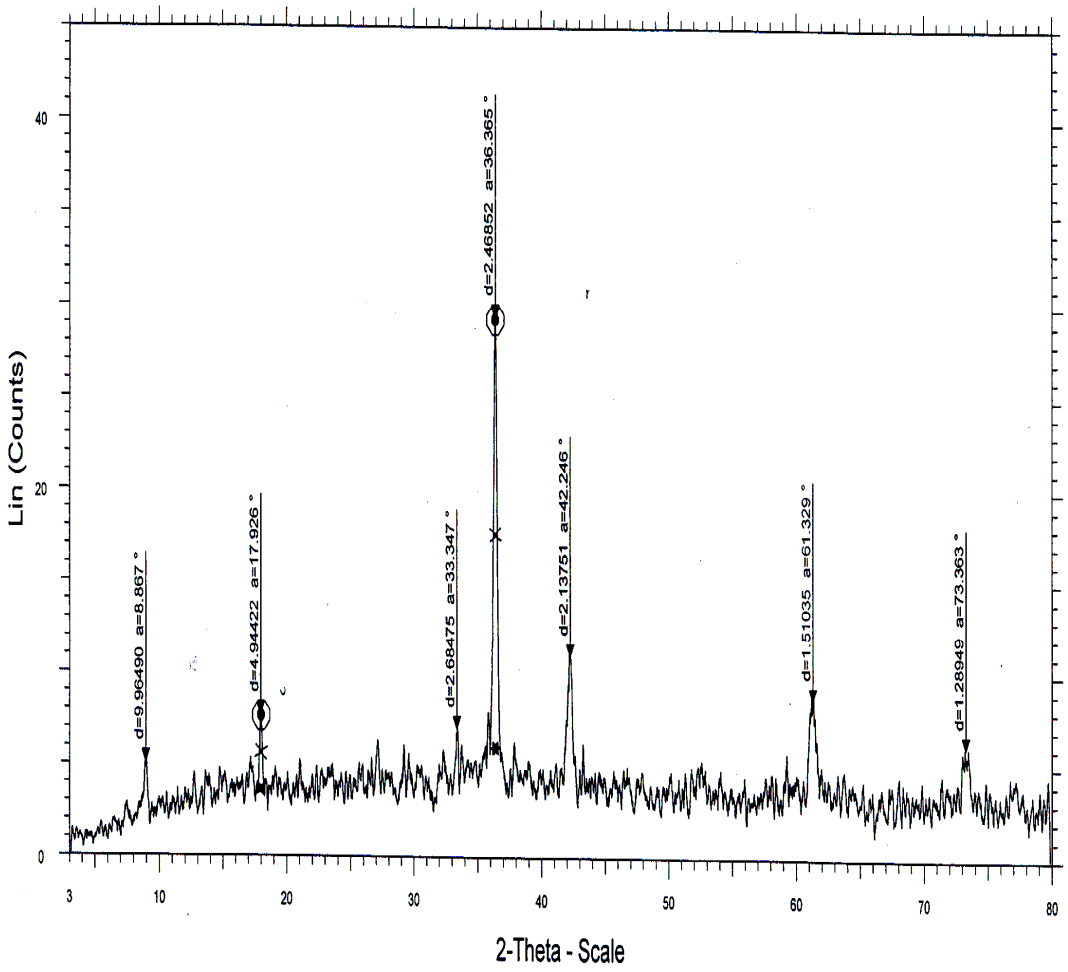

Figure 1. XRD pattern of obtained Cu_2O

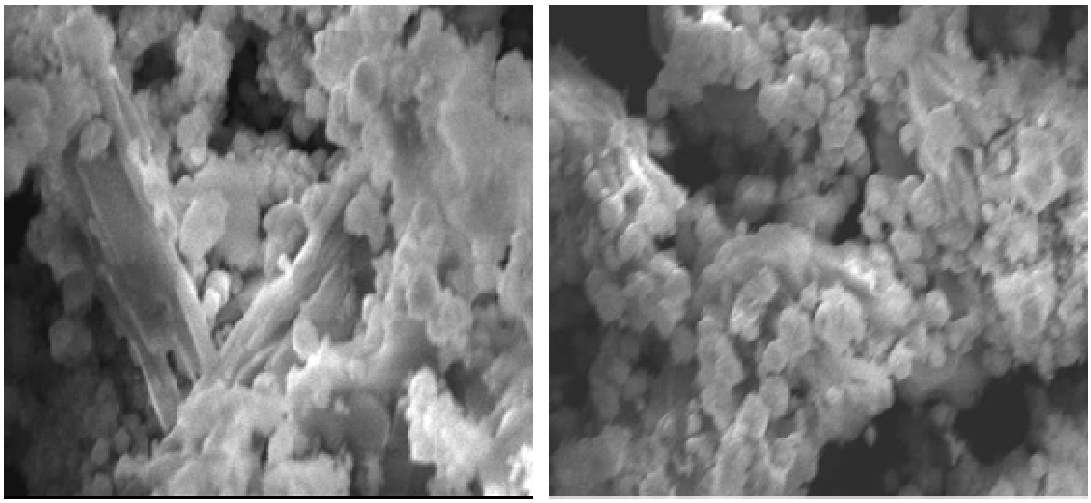

Figure 2. SEM images of as prepared Cu_2O nanoparticles

Table 1- Effect of reaction time

Sample No.	Concentration	Reaction temperature(°C)	Time duration(Hours)	Size of the particles (nm)
Sample 1	50ml PEG+3gCA	360	2	32-42
Sample 2	50mlPEG+3g CA	360	3	28-36
Sample 3	50mlPEG+3g CA	360	4	30-41
Sample 4	50mlPEG+3g CA	360	5	26-38
Sample 5	50mlPEG+3g CA	360	6	37

Table 2 - Effect of Temperature

Sample No.	Concentration	Reaction temperature (°C)	Time duration (Hours)	Size of the particles (nm)
Sample 6	50mlPEG+3gCA	100	5	21-22
Sample 7	50mlPEG+3gCA	150	5	20-21
Sample 8	50mlPEG+3gCA	200	5	18-22
Sample 9	50mlPEG+3gCA	250	5	17-19
Sample 4	50mlPEG+3gCA	360	5	26-38

Table 3 - Effect of reaction Concentration

Sample No.	Concentration	Reaction temperature(°C)	Time duration (Hours)	Size of the particles (nm)
Sample 10	50mlPEG+2g CA	250	5	33
Sample 11	100mlPEG+2gCA	250	5	33-49
Sample 9	50mlPEG+3g CA	250	5	17-19
Sample 12	100mlPEG+3gCA	250	5	57
Sample 13	150mlPEG+3gCA	250	5	45

ACKNOWLEDGEMENTS

This work was financially supported by Shri Vijayalakshmi Charitable Trust, Coimbatore. We would like to thank Sophisticated Test and Instrumentation Centre (STIC), Cochin for recording XRD and PSG College of Technology, Coimbatore for recording XRD and SEM.

REFERENCES

1. T. Kosugi, S. Kaneko, *J. Am. Ceram. Soc*. 81, 3117 (1998).
2. J.M. Zuo, M. Kim, M. O'Keeffe, J.C.H. Spence, *Nature* 401, 49 (1999).
3. J. Ling, J.P. Wolfe, *Phys. Rev. Lett*. 71, 1222 (1993).
4. S. Ikeda, T. Takata, T. Kondo, G. Hitoki, M. Hara, J.N. Kondo, K. Domen, H. Hosono, H. Kawazoe, A. Tanaka, *Chem. Commun*. 20, 2185 (1998).
5. M. Hara, T. Kondo, M. Komada, S. Ikeda, K. Shinohara, A. Tanaka, J. N. Kondo, K. Domen, *Chem. Commun.* 3, 357 (1998).
6. I. Lybinetsky, S. Thevuthasan, D.E. McCready, D.R. Baer, *J. Appl. Phys*. 94, 7926 (2003).
7. Y. T. Chen, W. Zhang, B. Z. Zhou, X. Q. Xu, Y. Q. Fan, Y. J. Guo, Z. X. Zhang, *Chinese Chemical Letters* 16, 245 (2005).
8. P. Poizot, S. Laruelle, S. Grugeon, L. Dupont, J. M. Taracon, *Nature* 407, 496 (2000).
9. M.Y. Shen, T. Yokouchi, S. Koyama, T. Goto, *Phys. Rev. B* 56, 13066 (1997).
10. W. Shi, K. Lim, X. Liu, *J. Appl. Phys*. 81, 2822 (1997).
11. J. Kong, N.R. Franklin, C. Zhou, M.G. Chapline, S. Peng, K. Cho, H. Dai, *Science* 287, 622 (2000).
12. M. Law, H. Kind, B. Messer, F. Kim, P. D. Yang, Angew. *Chem. Int. Edn*. 41, 2405 (2002).
13. Y.L. Wang, X.C. Jiang, Y.N. Xia, *J. Am. Chem. Soc.* 125, 16176 (2003).
14. A. Kolmakov, M. Moskovits, *Annu. Rev. Mater. Res*. 34, 151 (2004).
15. C. M. Lieber, Z.L. Wang, *MRS Bull*. 32, 99 (2007).
16. J.K. Barton, A. A. Vertegel, E. W. Bohannan, J. A. Switzer, *Chem. Mater*. 13, 952 (2001).
17. L.M. Huang, H. T. Wang, Z. B. Wang, et al. *Chem. Mater*. 14, 876 (2002).
18. S. Deki, K. Akamatsu, T. Yano, A. Kajinami, *J. Mater. Chem.* 8,1865 (1998).
19. R.V. Kumar, Y. Mastai, Y. Diamant, et al. *J. Mater. Chem*. 11, 1209 (2001).
20. H. Yanagimoto, K. Akamatsu, K. Gotoh, et al. *J. Mater. Chem*. 11, 2387 (2001).
21. S.J. Chen, X.T. Chen, L.H. Lia, et al. *J. Cryst. Growth* 246, 169 (2002).

22. Y. J. Dong, Y. D. Li, A. L. Cui, et al. *J. Colloid Interface Sci*. 243, 85 (2000).
23. W. Z. Wang, G. H. Wang, X. S. Wang, *et al. Adv. Mater*.14, 67 (2002).
24. S. Venkatachalam, D. Mangalaraj, S. K. Narayandass, R. Kesavamoorthy, P. Magudapathy, B. Sundaravel, S. Kalavathi, K. G. M. Nair, *Semicond. Sci. Technol*. 21, 1661 (2006).